

Performance Monitoring Protocol for FT-IR Systems

1 Scope

This document addresses the quality assurance/quality control (QA/QC) performance monitoring of the Fourier Transform Infrared (FT-IR) spectrometer systems utilized by personnel in the Trace Evidence Unit (TEU) and Scientific and Biometrics Analysis Unit - Trace (SBAU-Trace). Identification of generic polymeric group and classification of polymeric sub-group is conducted using the FT-IR system(s) identified below or an equivalent or better system. The performance of the system(s) must be monitored in order to verify that the instrument is producing reliable and reproducible results.

2 Equipment/Materials/Reagents

- Fourier Transform Infrared (FT-IR) Spectrometer with Microscope
Accessory: Thermo Nicolet 6700 or Nicolet is50 with Continuum microscope accessory, or equivalent or better system
- Liquid nitrogen
- Dewar flask
- Polystyrene standard: 1.5mil (38 micron) matte-finish film mounted on a card (Traceable and/or non-traceable. See Section 3.1)
- Standards wheel in Nicolet 6700 or Nicolet is50 spectrometer bench: 1.5mil (38 micron) matte-finish NIST traceable polystyrene standard and 1.0mil Schott NG11, National Physical Laboratory (NPL) traceable optical glass reference installed within the bench
- Pinhole slide: Slide containing a metal disk with a 100 micron pinhole, an open hole approximately 11mm in diameter, and a 14mm diameter gold mirror (for Continuum microscopes)

3 Standards and Controls

3.1 Daily Performance Standard

A 1.5mil polystyrene standard is analyzed as the performance standard to assess operating performance, wavenumber assignment, and continued integrity of the system. This analysis must be conducted each day the instrument is to be used before beginning casework analysis. The polystyrene standard used for this procedure requires no preparation. It is recommended by Thermo Scientific that the standard be replaced if showing signs of wear or if analysis of the standard does not produce the expected results. Newly acquired polystyrene standards that are accompanied by a statement of traceability to a NIST sample require no further testing before use. Non-serialized polystyrene standards that are not accompanied by a statement of traceability to a NIST sample require a comparative analysis with a traceable polystyrene

standard. This can be accomplished by comparison of spectral data to a spectrum obtained from a traceable, serialized polystyrene standard. The newly acquired polystyrene standard must also meet the Decision Criteria outlined in Section 5.3. If the newly acquired standard meets the aforementioned requirements, it can be used as a daily performance standard. If it does not meet the requirements, it will not be used as a daily performance standard. Spectral data produced during the acceptance process will be printed and stored within the TEU or SBAU-Trace FT-IR binder for the instrument it will be utilized with.

3.2 Performance Standards for Troubleshooting

The polystyrene standards on the standards wheel installed in the bench are analyzed during the Val-Q/ValPro quality assurance/quality control evaluations to verify that the components within the bench are performing as expected. (See Section 2)

The 1.5mil polystyrene standard that is used as the daily performance standard is also used to evaluate the Continuum microscope accessory as needed during troubleshooting.

The standards used for this procedure require no preparation. It is recommended that they be replaced if showing signs of wear or if analysis of the standards does not produce the expected results.

4 Sampling

Not applicable.

5 Procedures

5.1 Daily Performance Check

The following steps are to be performed each day the instrument is to be used before beginning casework analysis. The appropriate information will be recorded in the instrument logbook.

- a. Cool the detector by filling the internal Dewar with liquid nitrogen.
- b. Load the appropriate microscope transmission method, and verify the following parameters:
 - Mode = Transmission
 - Number of scans = 128
 - Resolution = 4
 - Scan range = 650-4000cm⁻¹ (wavenumbers)

- c. Collect a sample spectrum of the 1.5mil polystyrene standard followed by a background spectrum of air (empty stage).
- d. Use “Find Peaks” to label the major peaks. Evaluate the results using the Decision Criteria tabulated in Section 5.3.
- e. Save the spectrum of the polystyrene standard in the appropriate electronic folder.
- f. If the results are acceptable, the instrument may be used for casework. If the results are not acceptable, the polystyrene standard will be re-analyzed. If the results are still not acceptable, an out-of-service sign will be placed on the instrument, the appropriate log entry made, and the Technical Leader (TL) or SBAU - Instrumentation Operations group, contacted. The TL or SBAU - Instrumentation Operations group, will be responsible for ensuring the instrument is brought back into service. Any adjustments made will be recorded in the appropriate logbook.
- g. The results of the polystyrene spectral analysis are acceptable if the following four peaks are within +/- 4 cm^{-1} of the expected values (ASTM 1421-99 (2015)). Passing results will be recorded in the instrument logbook, when appropriate. If the peak values are not within the acceptable range, see section 5.2 for troubleshooting.

<u>Expected Value (cm^{-1})</u>	<u>Acceptable Range (cm^{-1})</u>
3025	3021 to 3029
1601	1597 to 1605
1028	1024 to 1032
906	902 to 910

5.2 FT-IR Bench and/or Microscope Accessory Troubleshooting

The following evaluations may be performed as needed based on system performance. The following evaluations can help to troubleshoot poor instrument performance or malfunction. Evaluation results will be recorded in the affected instrument’s logbook when appropriate.

5.2.1 Evaluation of the Bench Interferogram Signal

- a. Load the “Transmission ESP” method.
- b. On the “Bench” tab of “Experiment Setup”, monitor the interferogram signal under a gain of one (1.0).

- c. Record the peak-to-peak voltage of the interferogram in the instrument logbook. This value reflects the voltage being detected.
- d. If the signal value has dropped significantly from the previous evaluation, the beamsplitter can be automatically adjusted to improve the beam voltage throughput. Refer to the manufacturer's instrument manuals for further instructions.

5.2.2 Evaluation of the FT-IR Microscope Accessory Interferogram Signal

- a. Cool the detector with liquid nitrogen.
- b. Load the microscope transmission method.
- c. Set the objective and stage compensators to zero.
- d. Align and focus the microscope using the 100 micron pinhole slide.
- e. Monitor the interferogram signal under a gain setting of 1.0 on the Continuum.
- f. Record the peak-to-peak voltage of the interferogram in the instrument logbook.

5.2.3 Bench Evaluation

- a. Initiate the appropriate system validation/qualification program (Val-Q/ValPro) from within the instruments software (Omnicon or equivalent program). Run the Val-Q/ValPro validation to evaluate the performance of the bench. The pre-programmed software will automatically initiate the use of the internal standards which are installed within the bench of Thermo Scientific brand FT-IRs. (See Section 2)
- b. Evaluate the validation report. The results of each test will be listed as pass/fail. If any tests fail, follow the prompts within the software for bench alignment, then conduct a second evaluation with Val-Q/ValPro. If the results are acceptable, save the report in the appropriate electronic file. Print a copy of the report and place it in the appropriate binder
- b. If the results are still not acceptable, place an out-of-service sign on the instrument, make the appropriate log entry, and contact the TL or SBAU - Instrumentation Operations group. The TL or SBAU - Instrumentation Operations group is responsible for ensuring the instrument is brought back into service. Any adjustments made will be recorded in the appropriate logbook.

5.2.4 Continuum Microscope Evaluation

See Section 5.1.

6 Calculations

Not applicable.

7 Measurement Uncertainty

Not applicable.

8 Limitations

Only properly trained personnel will perform the duties involved in the operation, maintenance, and/or troubleshooting of this instrument.

9 Safety

Standard precautions for the handling of liquid nitrogen to include lab coat, goggles, and cryogenic gloves will be taken. Personal protective equipment (at a minimum, a laboratory coat and gloves) will be used when handling any chemical. Refer to the FBI Laboratory Safety Manual for the proper handling and disposal of all chemicals. No specific hazards are associated with the microscopy techniques performed. Universal precautions will be followed.

10 References

- ASTM 1421-99 (2015), Standard Practice for Describing and Measuring Performance of Fourier Transform Mid-Infrared (FT-MIR) Spectrometers: Level Zero and Level One Tests, American Society for Testing and Materials, West Conshohocken, PA.
- FBI Laboratory Safety Manual
- FBI Laboratory Chemistry Unit Instrument Operation & Support *Performance Monitoring Protocol (QA/QC) for the Nicolet FTIRs*
- Thermo Scientific, Nicolet™ FT-IR User's Guide, Thermo Electron Corporation, Madison, WI: 2004 (P/N 269-155800, Received with Nicolet™ 6700)

- Thermo Scientific, Nicolet™ Continuum™ Microscope User's Guide, Thermo Electron Corporation, Madison WI: 2006 (P/N 269-091804, Received with Nicolet™ 6700)
- Thermo Scientific, Nicolet™ is50 FT-IR Spectrometer User's Guide, Thermo Electron Corporation, Madison, WI: 2013-2014. Installed on is50 associated computer.
- Thermo Scientific, Nicolet™ Continuum™ Infrared Microscope User's Guide, Thermo Electron Corporation, Madison WI: 2007-2014. Installed on is50 associated computer.

Rev. #	Issue Date	History
4	10/02/2017	<p>Changed title to discipline/non-unit specific</p> <p>Section 1 - Added language denoting that protocol will be used by both TEU and SAU - Trace.</p> <p>Section 2 - Added Nicolet is50 instrument to list, Removed reference to Nicolet 670 bench.</p> <p>Section 3.1 - Added reference to SAU-Trace binder, changed to binder with instrument.</p> <p>Sections 5.1/5.2.3/5.3 - Added contacting SAU - Instrumentation Operations group if instrument is not within parameters and needs to be taken out of service.</p> <p>Section 10 - Updated references.</p>
5	02/03/2020	<p>Updated SBAU-Trace group name throughout.</p> <p>Updated in Section 5.1 e.</p> <p>Rearranged Decision Criteria to 5.1 g.</p> <p>Changed all reference to TEU Property Manager to TL.</p>

Approval

Redacted - Signatures on File

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Date: 01/31/2020

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QA Approval

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